15. *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*

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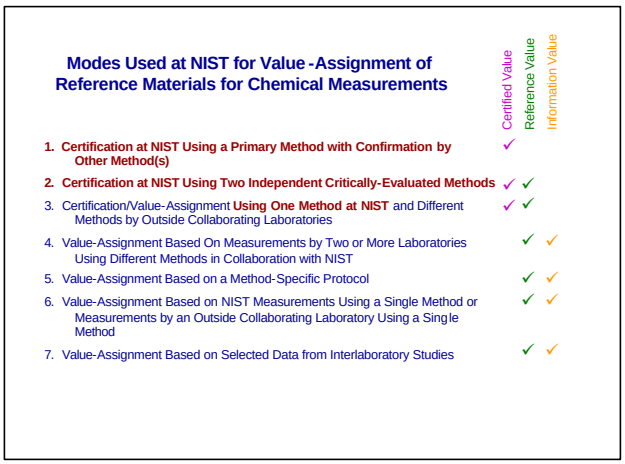
Objective: To develop a document which provides definitions of terms and descriptions of current practices used at NIST for value-assigning Standard Reference Materials (SRMs) for chemical composition and related properties.

Problem: NIST SRMs are used worldwide for providing quality assurance for chemical measurements. Chemical measurements are becoming increasingly important in international trade decisions and addressing healthcare, environmental and safety-related issues. There has been a recent proliferation of commercial and government-based reference material producers and the current ISO definitions for Reference Materials (RMs) and certified Reference Materials (CRMs). For example, a Reference Material is defined as *a material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials [ISO VIM: 1993, 6.13 [7]]*. A Certified Reference Material is defined as *a Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence [ISO VIM: 1993, 6.14]*. These definitions say nothing about the quality of the measurement science infrastructure that supports the value-assignment process for RMs and CRMs. This causes confusion for chemical measurement analysts worldwide as they attempt to establish increasingly required traceability for their chemical measurements.

Approach: The quality of assigned values for any CRM or RM is based on the existence and application of sound metrological principles and practices to the value-assignment process. It is with this basic premise that we have developed **NIST Special Publication 260-136**, "Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements". This document provides a complete description of the seven modes used at NIST to acquire analytical data for the value assignment of our SRMs and RMs for chemical measurements and links these modes to three data quality descriptors: NIST Certified Values, NIST Reference Values and NIST Information Values.

Results and Future Plans: A NIST Standard Reference Material® (SRM®) is a CRM issued by NIST that also meets additional NIST-specified certification criteria. NIST SRMs are issued with Certificates of Analysis or Certificates that report the results of their characterizations and provide information regarding the appropriate use(s) of the material. A *NIST Certified Value* is a value reported on an SRM Certificate/Certificate of Analysis for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by



NIST. Values are generally referred to as certified when Modes 1, 2, or 3 have been used for value-assignment and all the criteria for that mode are fulfilled. These three modes all require NIST measurements and oversight of the experimental design for the value-assignment process. The uncertainty associated with a certified value generally specifies a range within which the true value is expected to lie at a level of confidence of approximately 95 % if the sample is homogeneous. If significant sample heterogeneity is included, the uncertainty generally represents a prediction interval within which the true values of 95 % of all samples are expected to lie at a stated level of confidence.

A NIST Reference Value (formerly called Noncertified Value) is a best estimate of the true value provided on a NIST Certificate/Certificate of Analysis/Report of Investigation where all known or suspected sources of bias may not have been fully investigated by NIST. Reference values are generally determined using the following modes:

- Mode 2 or 3 is used when there is lack of sufficient agreement among the multiple methods.
- Modes 4, 5, or 6 are used when the intended use of the value by the measurement community does not require that it be a certified value.
- Mode 7 can be used in special cases, e.g., when results are obtained from another national metrology laboratory with whom NIST has historical comparability data for the method(s) used for the specific matrix/analyte combination.

The uncertainty associated with a NIST Reference Value may not include all sources of uncertainty and may represent only a measure of the precision of the measurement method(s).

A NIST Information Value is considered to be a value that will be of interest and use to the SRM/RM user, but insufficient information is available to assess the uncertainty associated with the value. Typically, the information value has no reported uncertainty listed on the certificate and has been derived from one of the following value-assignment modes:

- Results from modes 4, 5, 6, or 7 in which the intended use of the value by the measurement community does not require that it be a certified or reference value, e.g., information about the composition of the matrix such as the value of "total organic carbon" of a sediment material may be useful to the user in selecting an appropriate analytical method.
- The results from modes 4, 5, 6, or 7 lack sufficient information to assess the uncertainty.
- Results are provided from outside NIST as supplemental information on the SRM matrix and are not measurements typically made at NIST but may be of interest to the user.

NIST has met the chemical reference materials needs of U.S. industry and commerce for nearly 100 years. Coupled with the fast pace of technological change and greater measurement needs, the demand for additional quantities and additional specific varieties of reference materials has mushroomed. NIST, by itself, does not have the resources to provide SRMs (exact sample types, unique compound combinations, concentrations, etc.) to meet all these needs. Without a significant shift in paradigm, we will not be able to address future needs for reference materials, neither nationally nor internationally. The establishment of these seven modes for value assignment of NIST SRMs and RMs and communication of their linkage to the three quality descriptors (NIST Certified Values, NIST Reference Values and NIST Information Values) is a critical component of our strategy to produce an increasing number of SRMs and RMs through collaborative efforts with selected expert laboratories and other National Metrology Institutes/Standards Laboratories.

Publications:

"Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements", National Institute of Standards and Technology Special Publication 260-136, NIST Spec. Pub. 260-136 (November 1999).